ACCESSION NO. 77-061

COPY NO. 8

ISSUED TO: [signature]

PHILIP MORRIS CONFIDENTIAL

PHILIP MORRIS U.S.A.

RESEARCH CENTER

CHARGE NO. & TITLE: 1307 RECONSTITUTED TOBACCO DEVELOPMENT

TYPE REPORT: ○ ANNUAL ○ SEMIANNUAL ○ COMPLETION ⊗ SPECIAL

DATE: APRIL 1, 1977 PERIOD COVERED:

REPORT TITLE: RL DENITRATION
SEPARATE BURLEY PROCESSING

WRITTEN BY R. G. Uhl
R. G. UHL

SUPERVISED BY [signature]
G. GELLATLY

APPROVED BY [signature]
K. S. BURNS

DISTRIBUTION:

R. B. SELIGMAN
W. F. GANNON
K. S. BURNS
W. F. MUTTER
G. GELLATLY
S. HUTCHESON
E. T. OAKLEY
R. G. UHL

H. G. BURNLEY
J. A. GOULD
K. K. KHANDELWAL
W. G. LLOYD
A. R. PASQUINE

E. B. FISCHER
K. S. HOUGHTON
M. A. SERRANO
L. R. TURANO
CENTRAL FILES (6)

1003378789

KEYWORDS: DENITRATION, CRYSTALLIZATION, RL, CEL, NITROGEN OXIDE DELIVERY, POTASSIUM NITRATE, NITRATE NITROGEN, SEPARATE BURLEY PROCESSING

TABLE OF CONTENTS

1003378790

1003378791

I. SUMMARY

There is presently a strong desire to reduce the NO delivery of all PM cigarettes. It has been well established that there is a linear relationship between the nitric oxide (NO) delivery of a cigarette and the nitrate nitrogen (NO_3-N) content of it's filler. It has also been shown that approximately half of the nitrate nitrogen content of our blends comes from the burley stems in the RL and RCB sheet materials. Therefore, a process was developed in the RL Pilot Plant to selectively remove the NO_3-N (as potassium nitrate) from the concentrated extract liquor (CEL) generated in the RL process.[1] The CEL is chilled to crystallize potassium nitrate and then centrifuged to remove the salt. The CEL is denitrated from 0.8-1.2% NO_3-N to ~0.5% NO_3-N, providing a finished RL sheet with a NO_3-N content of ~0.4% versus 0.7-0.9% for the non-denitrated product.

Trials described in this report have shown that the bright tobacco portion of the blend is low in nitrate and if processed alone yields a CEL with a NO_3-N content of only ~0.2% without denitration. By separately extracting and denitrating the burley CEL, the combined CEL has a NO_3-N content of ~0.2-0.3%. This affords a 0.2% NO_3-N finished sheet or approximately a 50% nitrate reduction over denitration of the total blend.

Even though operating the plant in this manner (separate burley extraction) introduces some major process changes, it is our opinion that the additional nitrate reduction definitely justifies the adoption of this process at Park 500.

[1] RL Denitration Process, Acc. # 76-161.

II. PROCESS DESCRIPTION

A. Background

The nitric oxide (NO) delivery of Marlboro (360 ±40µg/cigt.) has been at the high end of the commercial cigarette range while the major competing brand, Winston, is at the low end of the range (280 ±30µg/cigt.). A relationship has been established relating cigarette gas phase NO with nitrate nitrogen (NO_3-N) content of the cigarette filler (Figure 1). Approximately half of the filler nitrate is present in the reconstituted tobaccos. This is due to high utilization of burley stems which can contain 7-25% potassium nitrate (KNO_3).

In the RL process the tobacco solubles are extracted with hot water and the remaining cellulosic fiber is formed into a sheet (base web) by paper making techniques. The tobacco solubles liquor (SEL) is concentrated from ~10% to ~50% solids (CEL) and is applied (with additives) to the base web to form a finished sheet.

B. Conventional Denitration of the URL Blend

The potassium nitrate in the tobacco feedstock is preferentially extracted by the hot water and is in essence wholly contained in the CEL. A process was developed in the RL Pilot Plant to remove the nitrate by chilling the CEL to just above the point of ice crystal formation (8-11 ^{0}F) to crystallize potassium nitrate. The cold liquor is then centrifuged to remove the crystallized salt. This process was recommended and accepted for installation in the commercial RL facility at Park 500 (ref. "RL Denitration Process", Acc. #76-161). The denitrated CEL has a NO_3-N content of ~0.5% (Figure 2) or about half the level in the initial CEL (0.8-1.3%).

C. Separate Denitration of Burley Tobacco

Analysis of samples of the individual unified blend feedstocks taken at the Blended Leaf Plant showed that the nitrate is largely concentrated in the burley components

1003378793

(Table I). The non-burley (or "bright") components of the unified blend (Table II) yield a CEL with a NO_3-N content of ~0.2% when separately processed or well below the denitrated blend CEL level of 0.5%. When the total blend is extracted, the bright and burley CELs are mixed and the nitrate content of bright CEL is raised while that of the burley CEL is diluted.

By separating the feedstock into bright and burley components (Table III), and maintaining separation of the CELs, a lower overall nitrate level is achieved (calculations are given in Appendix 1 a-e). The bright CEL remains at 0.2% NO_3-N. The undiluted burley CEL denitrates from a higher 1.8-3.0% NO_3-N to a lower level of ~0.4%. Burley can be denitrated to 0.4% due to it's lower freezing point (3-6 ^{0}F).

The net effect of separating the CELs is even more pronounced due to the fact that the burley portion, although comprising 43% of the feedstock, provides only 30% of the total CEL. There are three contributing reasons for this:

1. The burley portion is lower in hot water solubles (HWS), therefore providing less CEL than an equal weight of bright.
2. Burley is higher in fiber content causing more solubles to be carried forward with the base web (8-13% HWS).
3. The potassium nitrate is removed exclusively from the burley CEL further reducing it's contribution to the total CEL.

The additional nitrate removed by separate processing is presented graphically in Figure 3. The crosshatched area shows the additional nitrate removed by not increasing the bright CEL plus the potential additional removal by lower temperature centrifuging of the burley CEL.

Confirmatory tests were conducted in the RL Pilot Plant. Three tests were produced:

Test 201 - Denitration of separate burley

Test 202 - Whole blend denitration

Test 203 - Non-denitrated sheet

Test results (Table IV) showed a NO_3-N content in the non-denitrated sheet of 0.8%. This was reduced to 0.4% by conventional whole blend denitration and further reduced to 0.2% in the sheet made by denitration of the separate burley.

The two denitrated sheets were made into blended cigarettes (Model Y-79-1 MF) at the 13, 21 and 27% levels. The cigarette blend components were sampled and analyzed for % NO_3-N (Table V). These values were used to calculate predicted nitrate levels in the filler which agreed well with actual analysis of ripped cigarettes (Table VI). The cigarettes were smoked for gas phase analysis (Table VII) and those containing Test 201 sheet (denitration of separate burley) showed a reduction in NO of 40-60µg/cigarette when compared to whole blend denitration (Test 202).

1003378795

III. PROCESS CONSIDERATIONS FOR COMMERCIAL SCALE-UP

Separate burley processing could be installed on the single commercial line at Park 500 but would require duplication of pulpers, liquor tanks and associated pumps as well as installation of the planned third evaporator (spare at present rates). The planned installation of a second line provides numerous alternate modes of installation. These have been discussed with Park 500 management for their consideration for production design. One alternative would be to extract the burley on Line 1 where the denitration equipment is installed with the bright being extracted on Line 2. The extracted burley fiber from Line 1 press section would be metered to both lines' stock chests and likewise for the bright fiber from Line 2 press section. The (denitrated) CELs would be batch metered to both size preparation tanks or could be premixed. This type of installation makes the two lines interdependent since pulper downtime would affect both lines. Line independence can be achieved and downtime due to the pulpers halved by duplication of the pulpers and liquor tanks on both lines.

Regardless of the system installed, there are processing ramifications involved with handling the separated feedstocks. The main areas involved are as follows:

A. Blend Fraction Fiber Content (Pressing)

Park 500 has found the unified blend to require special handling (high pulper consistency) to achieve satisfactory press cake dryness as compared with the current RL blend. This can be due to four effects: high solubles content (fiber lubricity); high dust content and/or low stem content (all of which will be aggravated by feedstock separation), while the fourth, total press fiber content, will not be affected by separate processing. The aggravated conditions will exist in the bright portion which will exhibit:

1003378796

1. High solubles - The pilot plant has found solubles to impart a lubricity to the fiber which eases passage through the press drawing less amperage and yielding a wetter press cake. The bright solubles will be 50-55% while the burley will run 40-45%.

2. High dust content - The small particulate size of the dust portion may offer less resistance to pressing. The bright will be 53% dust while the burley is only 12%.

3. Low stem content - The pilot plant has found in processing all-stem (long fiber) feedstock that the presses draw more power in passing the stem. The bright portion will have a stem content of 47% (37% stem plus 10% winnowers) while the burley will be 88% stem.

The fourth consideration, i.e., the total amount of fiber in the presses, is estimated to be essentially the same for each line with separate processing (Table VIII). This is due to the higher blend percentage of the low fiber bright portion. Thus, the bright will be fed at a higher rate to the open throat presses which run at a partially starved condition. This should provide greater fiber compaction at the feed end of the press and increase cake dryness.

The combined effect is undeterminable and a production trial is planned. In addition, the low solubles and high fiber content of burley stems have given some pilot plant problems in pressing 100% burley stem stock. This occurs almost entirely at startup (fresh water extraction) when solubles and resultant lubricity are low. No problem is expected on the larger multi-stage commercial presses. The trial will also determine if there is any problem in sufficiently pulping the burley stem stock. The production batch pulpers are superior in design to the pilot plant model and should perform satisfactorily.

B. Blend Fraction Evaporator Load

The relative evaporator loads of the bright and burley portions show a higher evaporative requirement for the bright (Appendix 2). This is less than the 70-30 split of the CELs due to the higher solubles level in the bright SEL. In addition, if the burley is concentrated to the same refractive index solubles, its total solids content will be somewhat higher. The remaining offset is desirable since a higher percentage cleaning time will be required for the burley evaporators. This is due to the presence of potassium calcium sulfate monohydrate ($K_2Ca(SO_4)_2 \cdot H_2O$) which precipitates and causes fouling. The double salt, like the nitrate salt, is predominantly present in the burley stem fraction. Thus, more frequent sulfamic acid washing will be required. Fouling of the bright evaporators should likewise be reduced. This offset in evaporator load would be compensated for in the interdependent lines concept since Line 2 (bright portion) evaporators are planned to be larger than Line 1. (The third evaporator scheduled for Line 1 will provide a 50% evaporator capacity increase for a 25% planned sheet production increase.)

C. Crystallizer Heat Load

As has been noted previously, the burley CEL will be ∿30% of the recombined total CEL. Thus, the CEL cooling requirement for the separate burley at a two-line rate will be less than that required for whole blend denitration at a one-line rate. Even after accounting for the additional heat of crystallization of the extra potassium nitrate removed, the present Line 1 denitration equipment should suffice for both lines with some spare capacity (Appendix 3). There has been no evidence of double salt precipitation in the pilot plant crystallizer. Caking occurs in the evaporator outlet piping in both the commercial and pilot installations. No streamlining of this piping is recommended as precipitation here seems preferential to caking within the crystallizers.

D. Effect of Degree of Concentration

Pilot plant data shows a tendency toward lower nitrate left in burley CEL with higher solids content in the denitrated liquor (Figure 4). This can be explained by assuming nitrate to be soluble only in the aqueous portion of the CEL. Handbook values for potassium nitrate solubility, halved to approximate the 50% solids in CEL, approximate solubility in CEL (Figure 5). During production of the test sheets, the total solids of the burley CEL dropped by 10% due to denitration. This is reasonable since 2% NO_3-N (14% KNO_3) was removed (Table IX). The inference is that high nitrate in the burley stems will cause a higher total solids drop giving a denitrated CEL with a slightly higher nitrate content. However, this level would never exceed the blend denitration level of ∿0.5%. It is recommended that the burley CEL be concentrated to the maximum compatible with continuous operation.

E. Liquor Properties

The properties of the separate liquors were determined during the test run (Table IX). The burley CEL has a higher density initially but after denitration (during metering) it approximates the bright. The extraction liquor (SEL) solubles are normally higher in production and should approach 9% for the burley and 13% for the bright. The burley CEL and evaporator overheads are basic. The nicotine content of the overheads is less than 0.01% and the basicity can be accounted for by the ammonia present. Viscosities and colloid content were normal.

F. Crystal Size

Microphotographs of the nitrate crystals removed from the burley CEL show a larger size (100μ) than normally experienced with blend CEL (40μ) (Figure 6). The larger crystals, having less surface area, carry less solubles with them giving a purer potassium nitrate product. This larger crystal size carries over to the second stage separation (Figure 7). Purer crystals reduce losses, improve handling properties and increase product acceptability.

1003378799

IV. PRODUCT QUALITY

A. Physical Properties

The physical properties of the test sheets were determined (Table X). No appreciable difference was expected. The actual results are within the variation of normal pilot plant laboratory runs.

B. Subjective Evaluation

The 13% and 21% test cigarettes were evaluated in SEF (Appendix 4). The 13% cigarettes (actual Y-79-1 MF model) showed no significant difference between the control sheet (blend denitration) and the experimental sheet (burley denitration). The 21% cigarettes showed an overall preference for the control. These cigarettes, along with the 27% model, were also evaluated by the Flavor Development Group (Appendix 5). They found no obvious differences but detected a stemmy off-taste with both sheets at the higher levels. The Flavor Group has planned additional models for continued evaluation.

V. RECOMMENDATION

Separate burley denitration will reduce the NO_3-N content of RL by an additional 40-50% over whole blend denitration or an additional 25% of the undenitrated level. When compared to conventionally denitrated RL in cigarettes, denitration of separate burley affords an additional 40-60µg/cigarette reduction in NO. Since present projections indicate that the percentage of RL in our cigarettes can only increase, installation of separate burley denitration is recommended.

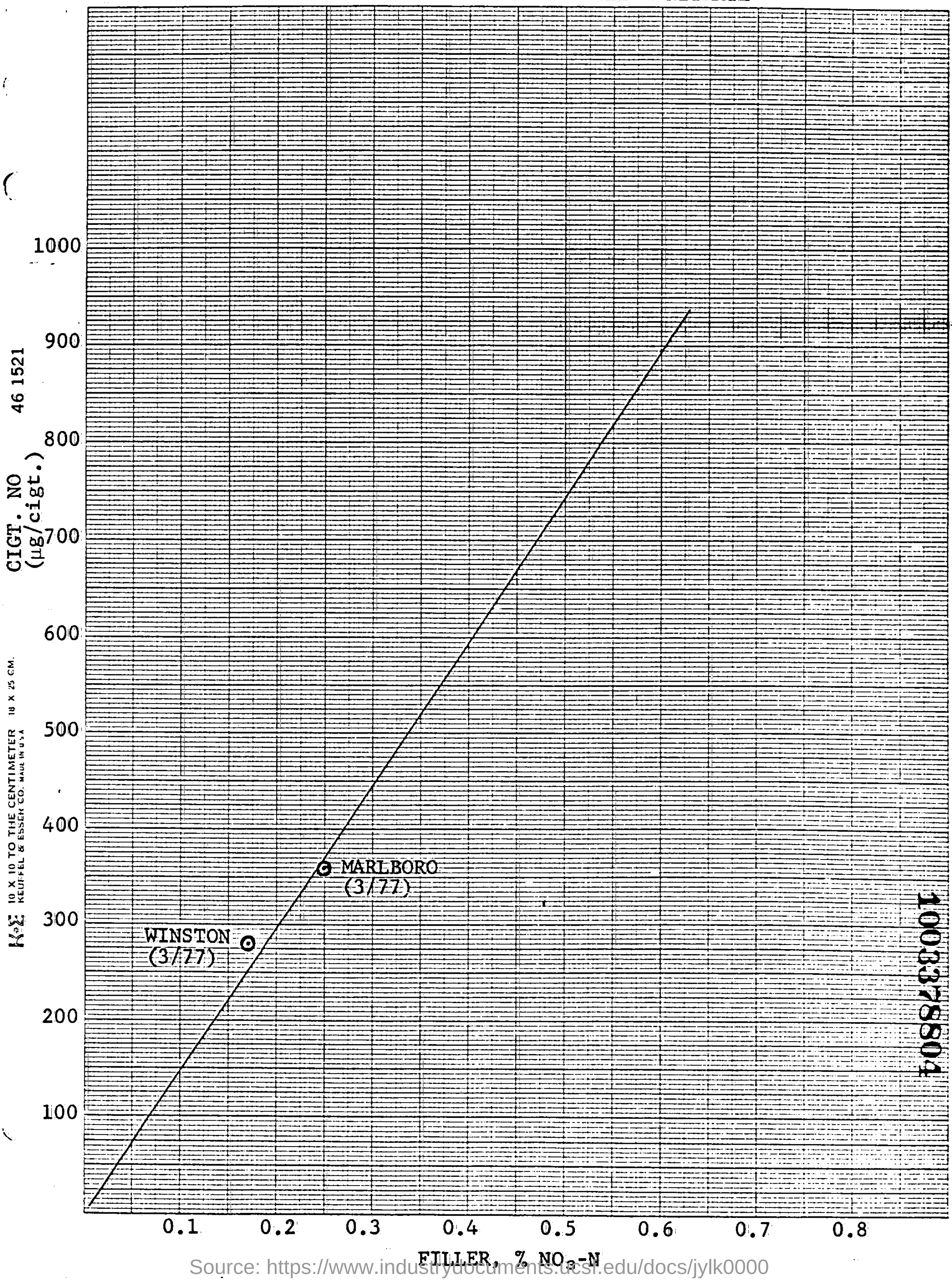
1003378801

VI. ACKNOWLEDGEMENTS

Development of the separate burley denitration process has been a team effort of the Reconstituted Tobacco Development Group under the supervision of Mr. G. Gellatly. The author wishes to acknowledge the technical advice of Mr. J. M. Czisny and of Mr. G. L. Wilkinson who conducted the pilot plant tests. We also express appreciation for the yeoman efforts of the General Analytical Group of Mrs. E. T. Oakley and for their investigative assistance with non-routine problems encountered in defining the process.

VII. FIGURES

FIGURE 1
CIGARETTE NO DELIVERY VS. FILLER NITRATE



46 1521

K&E 10 X 10 TO THE CENTIMETER 18 X 25 CM. KEUFFEL & ESSER CO. MADE IN U.S.A.

1003378804

FIGURE 2
NITRATE CONTENT OF CEL VS. TEMPERATURE

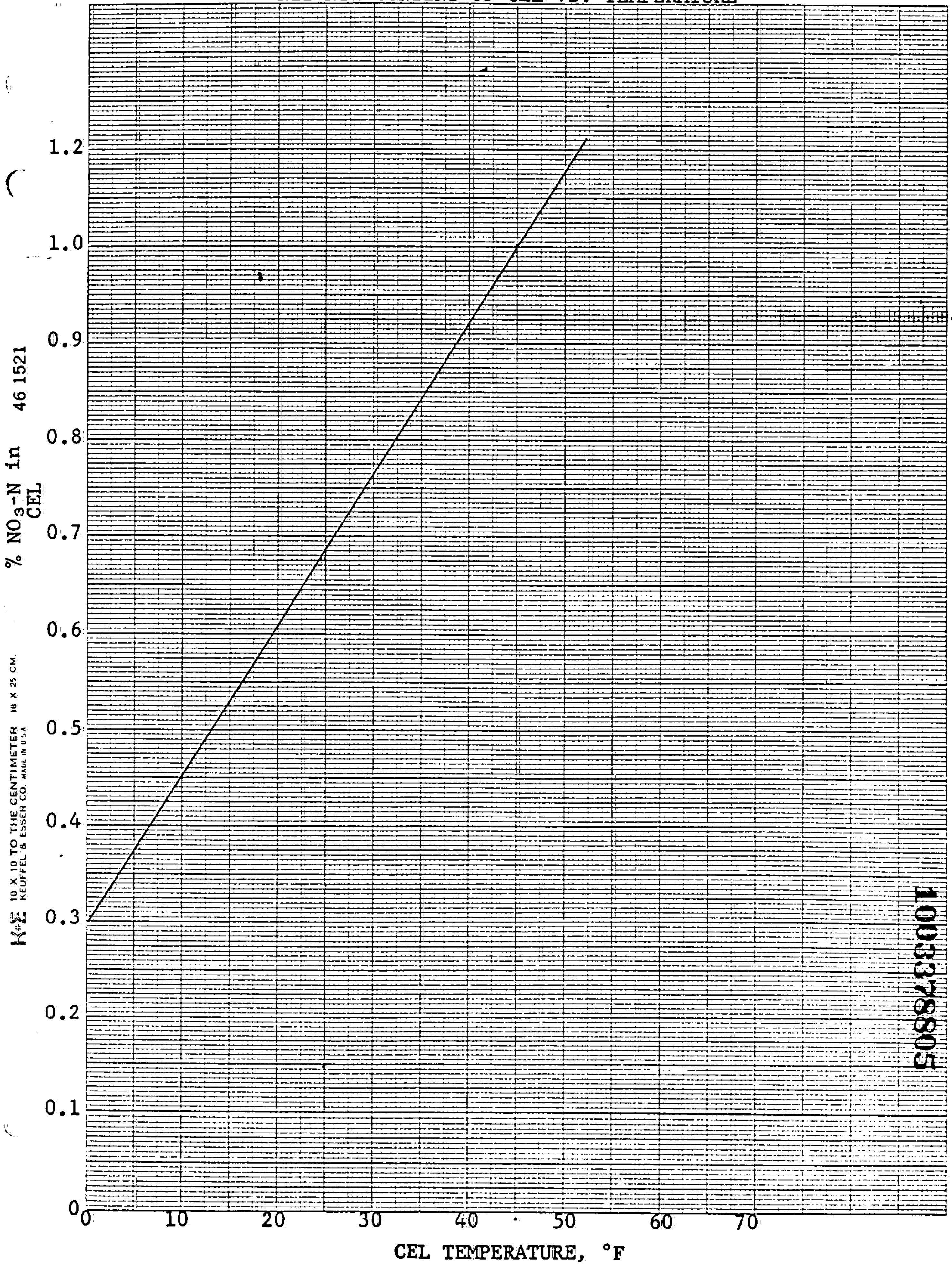
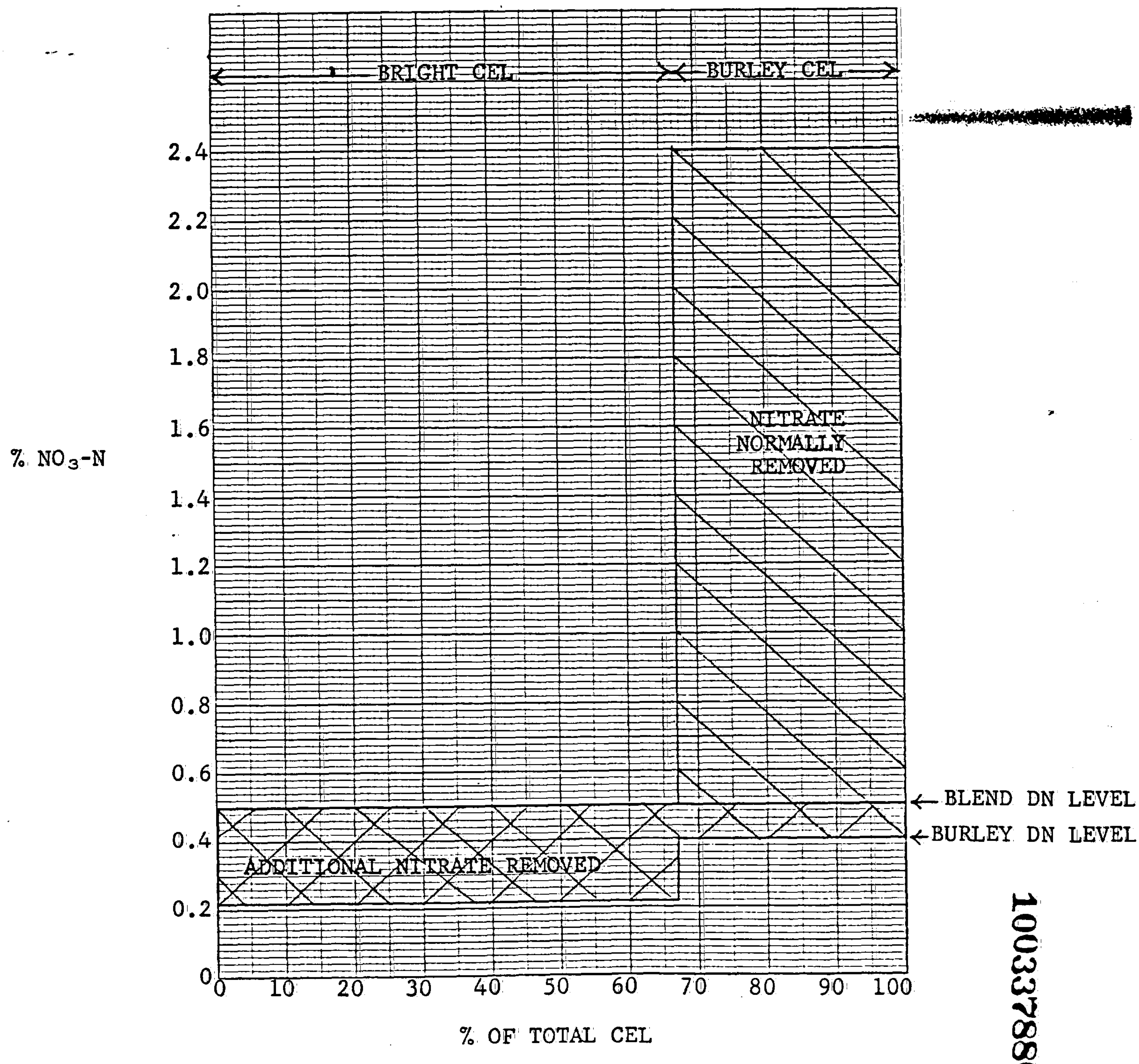


FIGURE 3

NITRATE CONTENT OF SEPARATE CELS



1003378806

FIGURE 4
NITRATE CONTENT OF BURLEY CEL VS. CONCENTRATION

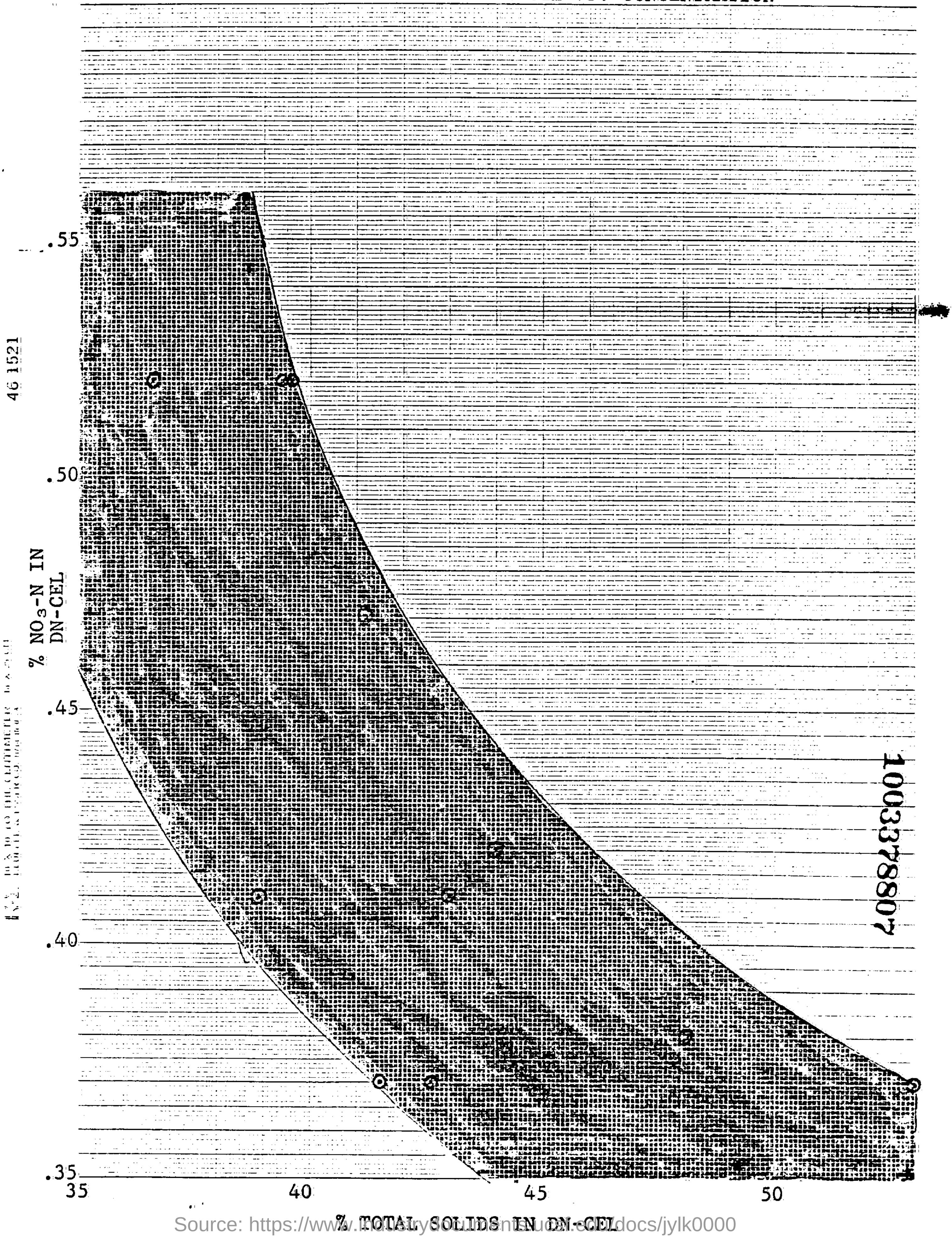
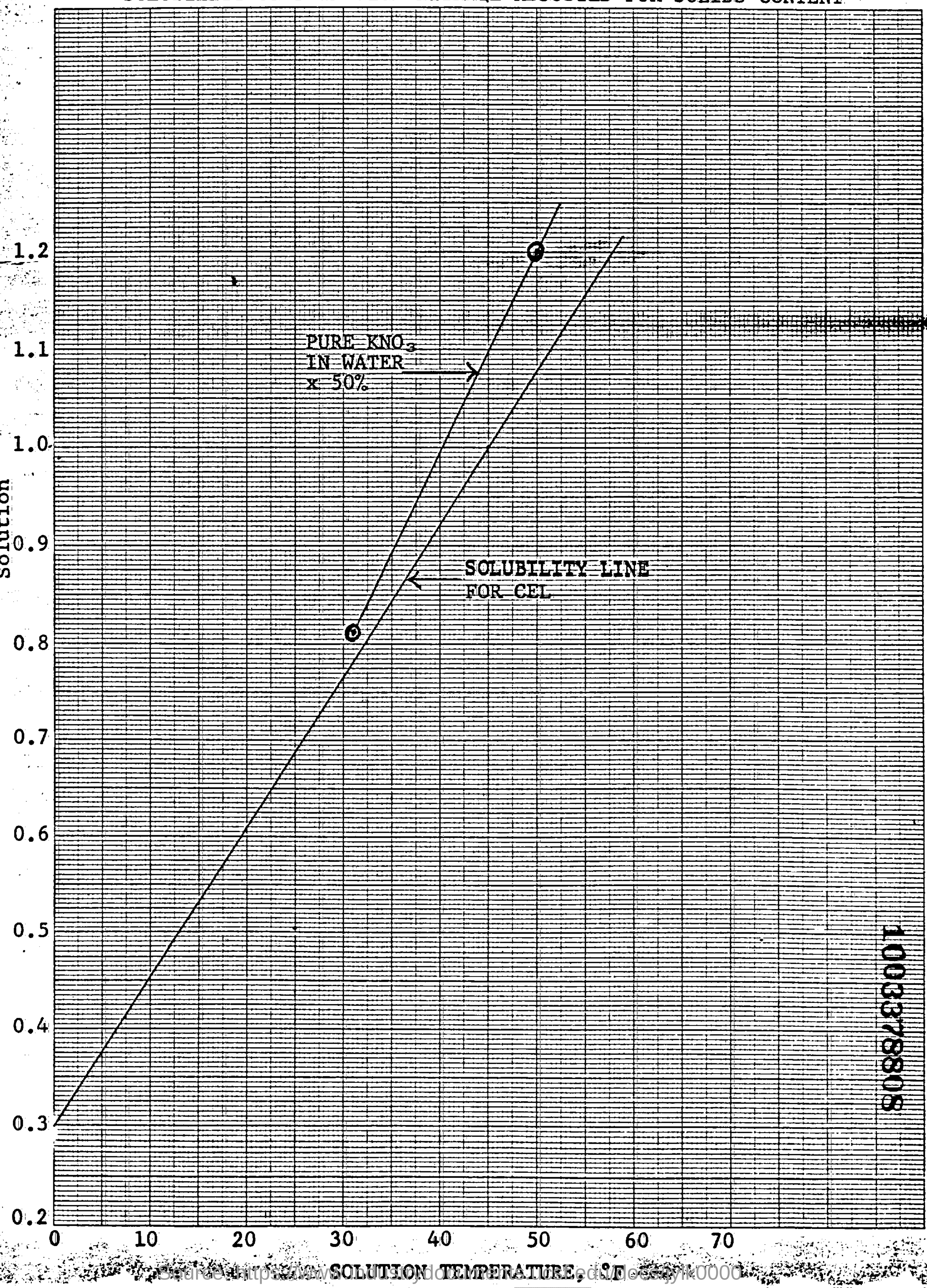


FIGURE 5
SOLUBILITY OF POTASSIUM NITRATE ADJUSTED FOR SOLIDS CONTENT

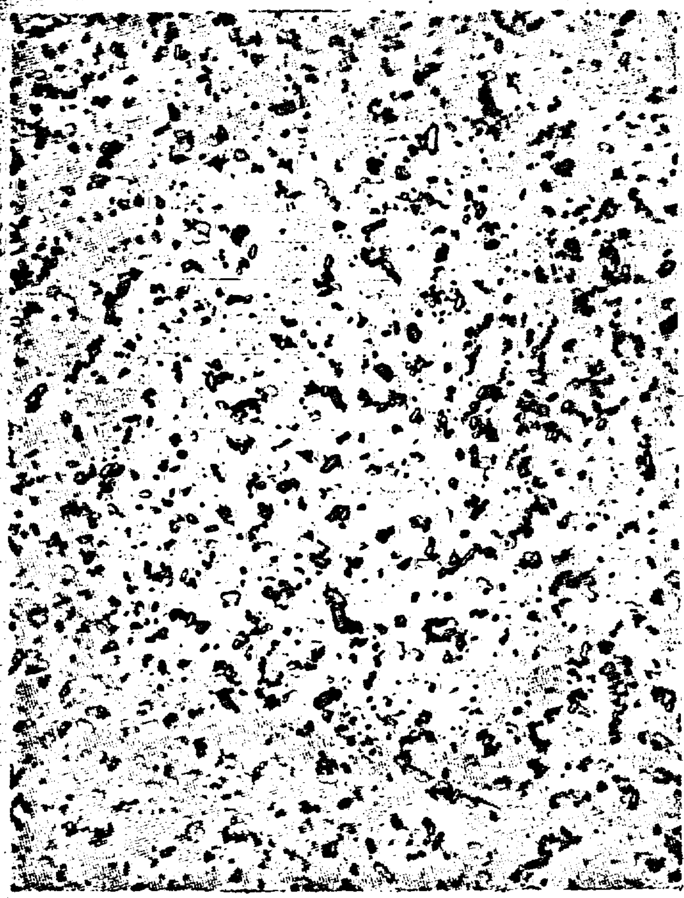


46 1521

1003378808

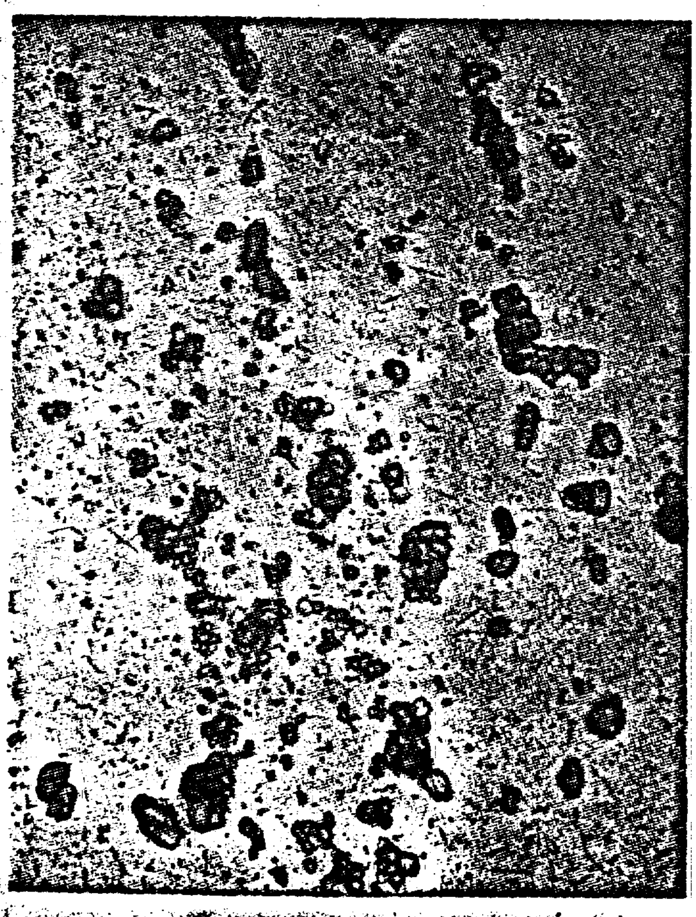
FIGURE 6

FIRST STAGE CRYSTALS (50x)



A. WHOLE BLEND DENITRATION

63% KNO_3 (WWB)
81% TOTAL SOLIDS



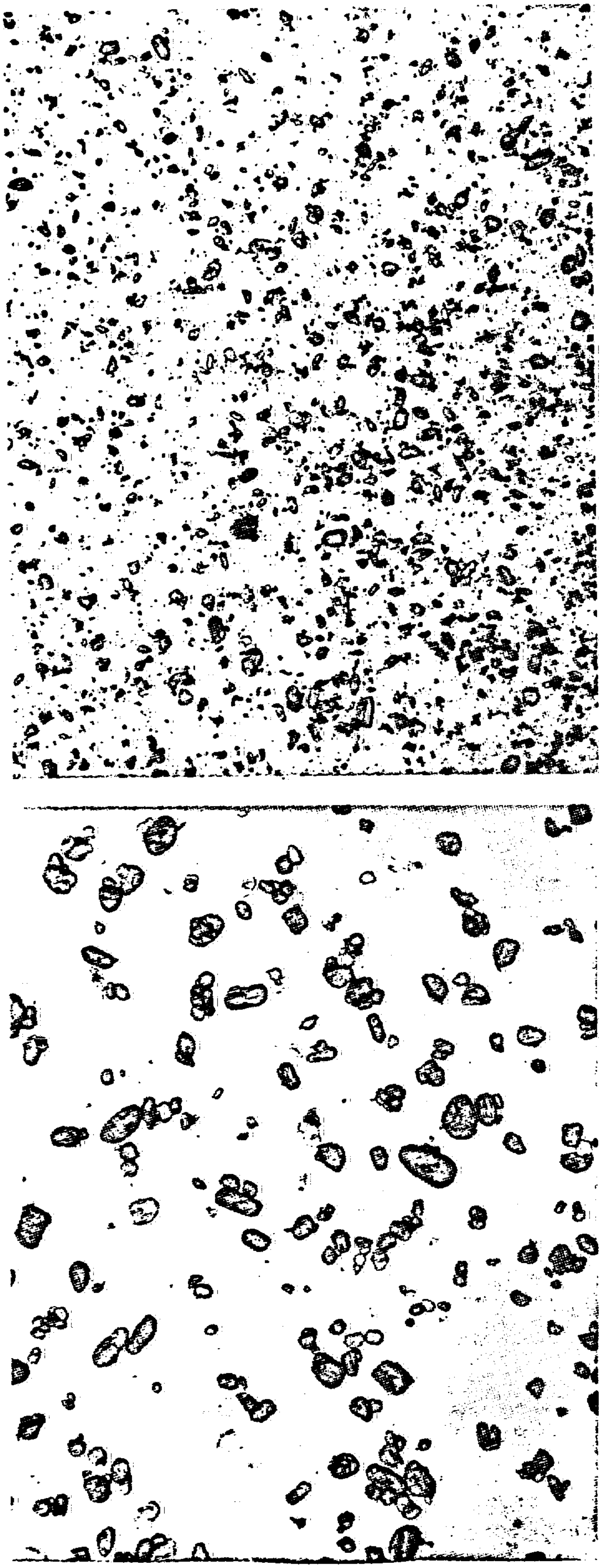
B. SEPARATE BURLEY DENITRATION

75% KNO_3 (WWB)
85% TOTAL SOLIDS

1003378809

FIGURE 7

SECOND STAGE CRYSTALS (50x)



A. <u>WHOLE BLEND DENITRATION</u>

85% KNO_3 (WWB)

9% OV

B. <u>SEPARATE BURLEY DENITRATION</u>

88% KNO_3 (WWB)

8% OV

1003378810

VIII. TABLES

TABLE I

NITRATE CONTENT OF BLEND COMPONENTS

	% NO_3-N	% HWS
BURLEY		
Burley Stems (KST)	1.95	44
Burley Stem Dust (KDM)	1.11	46
Burley Lamina Dust (KVF)	0.95	43
BRIGHT		
Bright Stem (VST)	0.14	56
Bright Lamina Scrap (BTBL)	ND	49
Bright Lamina Scrap (BVF)	ND	39
Bright Stem Dust (BDM)	ND	53
Turkish Lamina Scrap (TXS)	ND	55
CLASS TOBACCOS		
Class I (Primary Shorts)	0.28	54
Class II (Collector Dust)	0.17	38
Class III (Maker Dust)	0.28	52
Class IV (Winnowers)	0.49	56
Class VI (Menthol Dust)	0.25	53

(n = 8)

ND <.05

1003378812

TABLE II

UNIFIED BLEND

Dust Blend	% of Dust		% of URL Blend	
Bright				
BT Scrap & BVF	16.89			
BDM	7.50			
		24.39	10.00	
Burley				
KVF	9.15			
KDM	3.05			
		12.20	5.00	
Turkish				
Turkish Scrap	3.05			
		3.05	1.25	
FBP				
Class I	20.99			
Class II	0.27			
Class III	15.74			
Class V	0.59			
Class VI	7.07			
Class IV & VII	15.70			
		60.36	24.75	
		100.00		41.00
Bright Stem				
VST				21.00
Burley Stem				
KST				38.00
				100.00

TABLE III

COMPOSITION OF SEPARATE BLENDS

Non-Burley Portion

Dust Blend	% of Dust		% of Non-Burley Portion	% of URL Blend	
Bright					
BT Scrap & BVF	19.24				
BDM	8.54				
		27.78			
Turkish					
Turkish Scrap	3.45				
		3.47			
FBP					
Class I	23.91				
Class II	0.31				
Class III	17.93				
Class V	0.67				
Class VI	8.05				
Class IV & VII	17.88				
		68.75			
		100.00	63.16	36.00	
Bright Stem					
VST			36.84	21.00	
			100.00		57.00

Burley Portion	% of Burley Portion			
Burley Dust				
KVF	8.72			
KDM	2.91			
		11.63	5.00	
Burley Stem				
KST		88.37	38.00	
		100.00		43.00
				100.00

1003378814

TABLE IV

TEST SHEET ANALYSIS

Sheet Analysis (% DWB)	Blend DN (Test 202)	Burley DN (Test 201)
Nitrate Nitrogen	0.36	0.21
Hot Water Solubles	46	46
Phosphorous	0.77	0.71
Ammonia	0.82	0.61
Urea	3.07	3.32
Sorbic Acid	0.15	0.13
TEG	4.6	5.2

Control (undenitrated) sheet (Test 203): NO_3-N = 0.81%.

1003378815

TABLE V

NITRATE CONTENT OF CIGARETTE COMPONENTS

Blend Component	% NO_3-N (DWB)
Bright Strip	0.05
Burley Strip	0.53
Turkish Strip	0.06
ET	0.08
ES	0.35
URCB	0.42
URL (Burley DN)	0.19
URL (Blend DN)	0.33

NOTE: Samples taken in Semi-works during making of test cigarette blends.

1003378816

TABLE VI

NITRATE CONTENT OF TEST CIGARETTE FILLERS

Test Cigarette (Y79-1 MF)	Filler % NO_3-N (DWB)	
	Actual	Calculated*
13% URL (URCB, ES)		
Blend DN	0.24	0.23
Burley DN	0.22	0.22
21% URL (No URCB)		
Blend DN	0.23	0.23
Burley DN	0.20	0.20
27.5% URL (No URCB or ES)		
Blend DN	0.23	0.22
Burley DN	0.19	0.19

*Calculated from Table V and corrected for 12% casings.

1003378817

TABLE VII

NO DELIVERY OF TEST CIGARETTES

Test Cigarette (Y79-1 MF)	NO Delivery (μg/cigt.) Blend DN	Burley DN
13% URL (URCB, ES)	320	280
21% URL (No URCB)	300	235
27.5% URL (No URCB or ES)	310	250

(February, 1977, Marlboro - 360 μg/cigt.)

1003378818

TABLE VIII

BLEND FRACTION FIBER CONTENT

A. Basis: Feedstock Analysis (Table I), Blend Percentages (Tables II, III)

	Whole Blend	Burley Portion	Bright Portion
% of Feedstock	100	43	57
% HWS	49	44	52
% Fiber	51	56	48
% Stem[1]	65	88	47
% Dust	35	12	53

B. Blend Fraction Fiber Content

1) Burley Portion Fiber:

43% of blend x 56% fiber = 24% of blend = 47% of blend fiber

2) Bright Portion Fiber:

57% of blend x 48% fiber = 27% of blend = 53% of blend fiber

[1]Winnowers added to stem content and deleted from dust content (equal to 6% of whole blend, 10% of bright portion).

1003378819

TABLE IX

LIQUOR PROPERTIES

Process Stream	NO_3-N % WWB	R.I. Sol. %	Total Solids %	Susp. Solids Vol. %	pH	Colloid Vol. %	Viscosity Ambient (cps)	Viscosity Ambient (°F)	Viscosity 80°F (cps)	Specific Gravity
A. Whole Blend (Test 202)										
SEL	--	9.2	--	0.20	5.50	--	--	--	--	--
CEL	1.15	45.7	44.8	22	6.35	19	51	85	60	1.25
DN-CEL	0.36	44.4	41.5	19	6.60	21	101	48	55	1.22
B. Separate Blends (Test 201)										
1. Bright Portion										
SEL	--	10.4	--	0.26	5.25	--	--	--	--	--
CEL	0.18	45.4	41.5	32	5.80	15	79	82	98	1.20
2. Burley Portion										
SEL	--	6.7	--	0.10	6.60	--	--	--	--	--
CEL	2.41	47.2	50.7	47	8.15	19	85	91	108	1.32
DN-CEL	0.47	45.2	41.2	29	8.75	18	134	37	109	1.24
3. Combined										
(DN) CEL	0.22	44.7	41.1	29	6.20	21	118	76	234	1.22

NOTES:
1. Refractive Index solubles: All readings on standard sugar scale, Abbe bench refractometer.
2. Suspended Solids: Centrifuged as is for ten minutes.
3. Colloids: Diluted 2/1, centrifuged, decanted, sludge diluted 2/1, centrifuged.

1003378820

TABLE X

PHYSICAL PROPERTIES OF TEST SHEETS

Sheet Property	Blend DN (Test 202)	Burley DN (Test 201)
Thickness, mils	9.0	9.0
Equilibrium OV, %	15.2	14.1
Load Tensile, Kg/in	1.64	1.99
Elongation, %	2.6	2.5
Tensile Energy Absorption	2,368	2,640

1003378821

IX. APPENDICES

1. Calculated Nitrate Levels for Alternate Sheets
   a. Bright Fraction
   b. Burley Fraction
   c. Control (Undenitrated) Sheet
   d. Whole Blend Denitration
   e. Separate Burley Denitration

2. Blend Fraction Evaporation Load

3. Crystallizer Heat Load

4. SEF Evaluation
   a. 13% Cigarettes
   b. 21% Cigarettes

5. Flavor Development Group Evaluation

APPENDIX 1a

BRIGHT PORTION

Basis:

Feedstock:

52% HWS (separated as organic solubles & potassium nitrate)

0.25% NO_3-N = 1.81% KNO_3 (part of HWS)

45% CEL concentration

85% Reconstitution

Base web is 11% HWS.

Parts

48.00	Fiber	48.00	Fiber
1.81	Nitrate (Yields at 85% reconstitution)	1.54	Nitrate
50.19	Solubles	42.66	Solubles
100.00		92.20	

Base Web

$$\frac{48.00 \text{ Fiber}}{89\% \text{ Fiber}} = 53.93 \text{ Base Web Parts} = \begin{array}{l} 48.00 \text{ Fiber} \\ 5.93 \text{ Solubles} \end{array}$$

CEL

$$\begin{array}{rl} 42.66 \text{ Solubles} - 5.93 \text{ Base Web Solubles} = & 36.73 \text{ CEL Solubles} \\ + & \underline{1.54} \text{ Nitrate} \\ & 38.27 \text{ CEL Solids} \end{array}$$

$$\frac{38.27}{45\% \text{ Solids}} = 85.04 \text{ Parts CEL} = \begin{array}{l} 36.73 \text{ Solubles} \\ 1.54 \text{ Nitrate} \\ 46.77 \text{ Water} \end{array}$$

$$\frac{1.54 \text{ Nitrate}}{85.04 \text{ CEL}} = 1.81\% \text{ Nitrate} = 0.25\%\ NO_3\text{-N in Bright CEL}$$

1003378823

APPENDIX 1b

BURLEY PORTION

Basis:

Feedstock:

53% HWS
1.65% NO_3-N = 11.91% KNO_3
Denitrate to 0.4% NO_3-N.

Parts

57.00	Fiber		57.00	Fiber
11.91	Nitrate	(Yields at 85% reconstitution)	10.12	Nitrate
31.09	Solubles		26.43	Solubles
100.00			93.55	

Base Web

$$\frac{57.00 \text{ Fiber}}{89\% \text{ Fiber}} = 64.04 \text{ Base Web Parts} = \begin{array}{ll} 57.00 & \text{Fiber} \\ 7.04 & \text{Solubles} \end{array}$$

CEL

26.43 Solubles - 7.04 Base Web Solubles = 19.39 CEL Solubles
+ 10.12 Nitrate
29.51 CEL Solids

$$\frac{29.51}{45\% \text{ Solids}} = 65.58 \text{ Parts CEL} = \begin{array}{ll} 19.39 & \text{Solubles} \\ 10.12 & \text{Nitrate} \\ 36.07 & \text{Water} \end{array}$$

$$\frac{10.12 \text{ Nitrate}}{68.58 \text{ CEL}} = 15.43\% \text{ Nitrate} = 2.14\% \ NO_3\text{-N in Burley CEL}$$

DN-CEL

DN to 0.4% NO_3-N = 2.89% KNO_3
Water + Solubles = 100% - 2.89% = 97.11%

$$\frac{55.46 \text{ Water + Solubles}}{97.11\% \text{ Water + Solubles}} = \begin{array}{r} 57.11 \text{ Parts DN-CEL} \\ -55.46 \\ \hline 1.65 \text{ Nitrate} \end{array} = \begin{array}{ll} 19.39 & \text{Solubles} \\ 1.65 & \text{Nitrate} \\ 36.07 & \text{Water} \end{array}$$

$$\frac{1.65 \text{ Nitrate}}{57.11 \text{ CEL}} = 2.89\% \text{ Nitrate} = 0.40\% \ NO_3\text{-N in Burley DN-CEL}$$

$$\frac{19.39 \text{ Solubles} + 1.65 \text{ Nitrate}}{57.11 \text{ CEL}} = 36.8\% \text{ Solids in Burley DN-CEL}$$

1003378824

APPENDIX 1c

CONTROL (UNDENITRATED) SHEET

Basis: Bright Portion = 57%
Burley Portion = 43%
Finished Sheet = 13% additives analyzing as HWS

Bright Portion (Appendix 1a) x 57%:

30.74 Parts Base Web	=	27.36	Fiber
		3.38	Solubles
48.48 Parts CEL	=	20.94	Solubles
		0.88	Nitrate
		26.66	Water

Burley Portion (Appendix 1b) x 43%:

27.53 Parts Base Web	=	24.51	Fiber
		3.02	Solubles
28.20 Parts CEL	=	8.34	Solubles
		4.35	Nitrate
		15.51	Water

Combined:

58.27 Parts Base Web	=	51.87	Fiber
		6.40	Solubles
76.68 Parts CEL	=	29.28	Solubles
		5.23	Nitrate
		42.17	Water

CEL

$$\frac{5.23 \text{ Nitrate}}{76.68 \text{ CEL}} = 6.82\% \text{ Nitrate} = 0.95\% \ NO_3\text{-N}$$

Finished Sheet (F.S.)

58.27 Parts Base Web
+ 34.51 Parts CEL Solids
92.78 Parts Tobacco

At 13% Additives:

$$\frac{92.78 \text{ Tobacco}}{87\% \quad \text{Tobacco}} = 106.64 \text{ Parts F.S.}$$

$$\frac{106.64 \text{ F.S.} - 51.87 \text{ Fiber}}{106.64 \text{ F.S.}} = 51.4\% \text{ HWS in F.S. (DWB)}$$

$$\frac{5.23 \text{ Nitrate}}{106.64 \text{ F.S.}} = 4.90\% \text{ Nitrate} = 0.68\% \ NO_3\text{-N in F.S. (DWB)}$$

1003378825

APPENDIX 1d

WHOLE BLEND DENITRATION

Basis: Same as for undenitrated sheet (Appendix 1c). Denitrate to 0.5% NO_3-N.

$$58.27 \text{ Parts Base Web} = \begin{array}{l} 51.87 \text{ Fiber} \\ 6.40 \text{ Solubles} \end{array}$$

$$76.68 \text{ Parts CEL} = \begin{array}{l} 29.28 \text{ Solubles} \\ 5.23 \text{ Nitrate} \\ 42.17 \text{ Water} \end{array}$$

DN-CEL

Denitrate to 0.5% NO_3-N = 3.61% KNO_3
Water + Solubles = 100% - 3.61% = 96.39%

$$\frac{71.45 \text{ Parts Water + Solubles}}{96.39\ \%} = 74.13 \text{ Parts DN-CEL} = \begin{array}{l} 29.28 \text{ Solubles} \\ 2.68 \text{ Nitrate} \\ 42.17 \text{ Water} \end{array}$$

$$\text{Water + Solubles} - \frac{71.45}{2.68 \text{ Nitrate}}$$

$$\frac{2.68 \text{ Nitrate}}{74.13 \text{ CEL}} = 3.61\% \text{ Nitrate} = 0.50\%\ NO_3\text{-N in Whole Blend DN-CEL}$$

$$\frac{29.28 \text{ Solubles} + 2.68 \text{ Nitrate}}{74.13 \text{ CEL}} = 43.1\% \text{ Solids in Whole Blend DN-CEL}$$

Finished Sheet (F.S.)

$$\begin{array}{rl} & 58.27 \text{ Parts Base Web} \\ + & 31.96 \text{ Parts DN-CEL Solids} \\ \hline & 90.23 \text{ Parts Tobacco} \end{array}$$

At 13% Additives:

$$\frac{90.23 \text{ Tobacco}}{87\% \quad \text{Tobacco}} = 103.71 \text{ Parts F.S.}$$

$$\frac{103.71 \text{ F.S.} - 51.87 \text{ Fiber}}{103.71 \text{ F.S.}} = 50.0\% \text{ HWS in F.S. (DWB)}$$

$$\frac{2.68 \text{ Nitrate}}{103.71 \text{ F.S.}} = 2.58\% \text{ Nitrate} = 0.36\%\ NO_3\text{-N in F.S. (DWB)}$$

APPENDIX 1e

SEPARATE BURLEY DENITRATION

Basis: Same as for undenitrated sheet (Appendix 1c) except 43% of burley DN-CEL (Appendix 1b) is taken.

Burley DN-CEL x 43%:

24.56 Parts DN-CEL	=	8.34	Solubles
		0.71	Nitrate
		15.51	Water

Combined:

58.27 Parts Base Web	=	51.87	Fiber
		6.40	Solubles
73.04 Parts (DN) CEL	=	29.28	Solubles
		1.59	Nitrate
		42.17	Water

CEL

$$\frac{1.59\ \text{Nitrate}}{73.04\ \text{CEL}} = 2.18\%\ \text{Nitrate} = 0.30\%\ NO_3\text{-N in Combined (DN) CEL}$$

$$\frac{29.28\ \text{Solubles} + 1.59\ \text{Nitrate}}{73.04\ \text{CEL}} = 42.3\%\ \text{Solids in Combined (DN) CEL}$$

Finished Sheet (F.S.)

58.27 Parts Base Web
+ 30.87 Parts CEL Solids
89.14 Parts Tobacco

At 13% Additives:

$$\frac{89.14\ \text{Tobacco}}{87\%\ \text{Tobacco}} = 102.46\ \text{Parts F.S.}$$

$$\frac{102.46\ \text{F.S.} - 51.87\ \text{Fiber}}{102.46\ \text{F.S.}} = 49.4\%\ \text{HWS in F.S. (DWB)}$$

$$\frac{1.59\ \text{Nitrate}}{102.46\ \text{F.S.}} = 1.55\%\ \text{Nitrate} = 0.21\%\ NO_3\text{-N in F.S. (DWB)}$$

1003378827

APPENDIX 2

BLEND FRACTION EVAPORATION LOAD

A. Evaporation Load/Lb. CEL

1. Burley Liquor:

CEL (45% conc.)	SEL (9% conc.)
0.45 lb. sol.	0.45 lb. sol.
0.55 lb. water	4.55 lb. water

Therefore, 4.55 - 0.55 = 4.0 lb. water evaporated/lb. burley CEL.

2. Bright Liquor:

CEL (45% conc.)	SEL (13% conc.)
0.45 lb. sol.	0.45 lb. sol.
0.55 lb. water	3.05 lb. water

Therefore, 3.05 - 0.55 = 2.5 lb. water evaporated/lb. bright CEL.

B. Blend Fraction Evaporation Load

Basis: Bright to burley CEL ratio taken as 2 to 1 (Appendix 1)

1. Burley Liquor:

$$\frac{4.0\text{ lbs. water evaporated}}{1.0\text{ lb. burley CEL}} \times \frac{1.0\text{ lb. burley CEL}}{3.0\text{ lbs. combined CEL}} =$$

$$1.33\ \frac{\text{lbs. water}}{\text{lb. combined CEL}} = 45\%\text{ of total load}$$

2. Bright Liquor:

$$\frac{2.5\text{ lbs. water evaporated}}{1.0\text{ lb. bright CEL}} \times \frac{2.0\text{ lbs. bright CEL}}{3.0\text{ lbs. combined CEL}} =$$

$$1.66\ \frac{\text{lbs. water}}{\text{lb. combined CEL}} = 55\%\text{ of total load}$$

APPENDIX 3

CRYSTALLIZER HEAT LOAD

Basis: Heat Load/Gal. Blend CEL

A. Whole Blend Denitration

Basis: Second stage centrifuge recycles 25% of the nitrate and 3% of the CEL.

$$\left(1.0 \text{ gal. CEL} + 0.03 \text{ gal. CEL recycle}\right) \times \frac{10.4 \text{ lbs. CEL}}{\text{gal. CEL}} \times \left(\frac{0.88 \text{ Btu}}{\text{lb. CEL-F}^\circ} \times \right.$$

$$(110-10)\text{F}^\circ + (100\% + 25\% \text{ recycle})\ (0.009 - 0.005)\ \frac{\text{lb. } NO_3\text{-N}}{\text{lb. CEL}} \times$$

$$\left. \frac{7.22 \text{ lb. } KNO_3}{\text{lb. } NO_3\text{-N}} \times \frac{153.5 \text{ Btu}}{\text{lb. } KNO_3}\right) = 1{,}000 \text{ Btu}$$

B. Separate Burley Denitration

Basis: Nitrate removed increases 50%; therefore, recycle streams increase 50%. Burley CEL taken to 5°F and denitrated from 2.4 to 0.4% NO_3-N.

$$\left(1.0 \text{ gal. CEL} + 0.05 \text{ gal. CEL recycle}\right) \times \frac{10.4 \text{ lbs. CEL}}{\text{gal. CEL}} \times \left(\frac{0.88 \text{ Btu}}{\text{lb. CEL-F}^\circ} \times \right.$$

$$(110-5)\text{F}^\circ + (100\% + 38\% \text{ recycle})\ (0.024 - 0.004)\ \frac{\text{lb. } NO_3\text{-N}}{\text{lb. CEL}} \times$$

$$\left. \frac{7.22 \text{ lbs. } KNO_3}{\text{lb. } NO_3\text{-N}} \times \frac{153.5 \text{ Btu}}{\text{lb. } KNO_3}\right) \times \frac{1.0 \text{ lb. burley CEL}}{3.0 \text{ lbs. combined CEL}} = 450 \text{ Btu}$$

Therefore, the heat load for burley CEL at a two-line rate would be 2 x 450 Btu = 900 Btu or 90% of Line 1 design.

NOTE: Line 1 centrifuges, etc., were designed to handle the crystal volume generated by a burley stem feedstock.

1003378829

APPENDIX 4a

SEF EVALUATION
13% CIGARETTES

TITLE: Y-79-1 MF w/pp #202 (13%) RL (WHOLE BLEND DENITRATION) CODED X6D7CLT "C" vs. Y-79-1 MF w/pp #201 RL (13%) (BURLEY ONLY DENITRATED CODED X6D7CLY "E"

SUMMARY: THE DIFFERENCES FOUND ON THE ATTRIBUTE QUESTIONS, PREFERENCE QUESTION, AND MEAN RATINGS WERE NOT STATISTICALLY SIGNIFICANT.

REQUESTER: R. G. UHL   CHARGE # 1307   N = 41

TEST METHOD: PAIRED COMPARISON

PURPOSE: TO DETERMINE DIFFERENCES IN SMOKE FLAVOR

PREPARATION: CRACKERS AND WATER

PROCEDURE: USING REDUCED LIGHTING AND ABOVE PREPARATION, JUDGES WERE GIVEN THE SAMPLES IN A BALANCED PRESENTATION. THEN THEY ANSWERED THE QUESTIONS ON THE ATTACHED BALLOT.

RESULTS:

	CONTROL X6D7CLT	EXPERIMENTAL X6D7CLY	NO DIFFERENCE	PROBABILITY
1. WHICH IS HARSHER (MORE INHALATION IMPACT)?	8	17	16	NS
2. WHICH HAS MORE TOTAL TASTE?	7	16	18	NS
3. WHICH HAS AN OFF-TASTE?	5	7	28	NS
4. WHICH IS MORE SPICY (MORE PEPPERY OR STINGING TO MOUTH AND NASAL AREAS)?	11	17	13	NS
5. WHICH IS SWEETER?	13	10	18	NS
6. WHICH IS MORE BITTER?	5	11	24	NS
7. WHICH IS MORE TOBACCO-LIKE IN FLAVOR?	9	10	22	NS
8. WHICH IS HOTTER?	5	13	23	NS
9. WHICH CIGARETTE DO YOU PREFER?	12	17	12	NS

OVERALL ACCEPTABILITY		
	C	E
M	4.34	4.48
T	0.73	
P	NS	

SIGNED: Archie Willi

DATE RAN 3/16/77
DATE COMPLETED: March 16, 1977

1003378830

APPENDIX 4b

SEF EVALUATION
21% CIGARETTES

TITLE: Y-79-1 MF W/20.75% RL PP=202 (WHOLE BLEND DENITRATED) CODED X6D7CLU "C" VS Y-79-1 MF W/20.75% RL PP=201 (BURLEY ONLY DENITRATED) CODED X6D7CLV "E"

SUMMARY: THE CONTROL CIGARETTE CODED X6D7CLU WAS FOUND TO HAVE A HIGHER MEAN RATING ON THE NINE-POINT ACCEPTABILITY SCALE ($p<.02$)

REQUESTER: R. G. UHL CHARGE # 1307 N: 46

TEST METHOD: PAIRED COMPARISON

PURPOSE: TO DETERMINE DIFFERENCES IN SMOKE FLAVOR

PREPARATION: CRACKERS AND WATER

PROCEDURE: USING REDUCED LIGHTING AND ABOVE PREPARATION, JUDGES WERE GIVEN THE SAMPLES IN A BALANCED PRESENTATION. THEN THEY ANSWERED THE QUESTIONS ON THE ATTACHED BALLOT.

RESULTS:

	CONTROL X6D7CLU	EXPERIMENTAL X6D7CLV	NO DIFFERENCE	PROBABILITY
1. WHICH IS HARSHER (MORE INHALATION IMPACT)?	16	20	10	NS
2. WHICH HAS MORE TOTAL TASTE?	14	16	15	NS
3. WHICH HAS AN OFF-TASTE?	10	15	19	NS
4. WHICH IS MORE SPICY (MORE PEPPERY OR STINGING TO MOUTH AND NASAL AREAS)?	17	19	9	NS
5. WHICH IS SWEETER?	16	10	20	NS
6. WHICH IS MORE BITTER?	14	15	17	NS
7. WHICH IS MORE TOBACCO-LIKE IN FLAVOR?	16	14	15	NS
8. WHICH IS HOTTER?	12	19	15	NS
9. WHICH CIGARETTE DO YOU PREFER?	23	15	8	NS

OVERALL ACCEPTABILITY	C	E
M	4.52	3.89
T	2.48	
P	<.02	

SIGNED: Archie Williams

DATE RAN: 3/17/77

DATE COMPLETED: MARCH 18, 1977

1003378831

APPENDIX 5
FLAVOR DEVELOPMENT GROUP EVALUATION

PHILIP MORRIS U. S. A.

INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

To: Mr. R. G. Uhl Date: March 21, 1977

From: J. W. Swain

Subject: Subjective Screening of Cigarettes with URL with Whole Blend CEL Compared to Burley Portion Denitrated

Comparisons were made between URL #201 and #202 at 13.0, 20.75 and 27.5% blend levels. Comments indicated that the 13 and 20.75% levels smoked more uniform puff to puff than the 27.5% blends; therefore, differences in flavor were more difficult to judge on the latter. No obvious differences in flavor were shown between the types of denitrated URL although the higher blend levels were described as having stemmy off tastes.

In order to test the proposed '79 Marlboro, URL-SD should be evaluated after denitration of the whole blend CEL versus only the Burley portion of CEL. The acceptability of the 27.5% blends should then be evaluated in larger scale testing of the '79 Marlboro with and without URCB.

/ec

cc: F. L. Daylor
H. L. Spielberg
G. Gellatly

J. W. Swain

1003378832